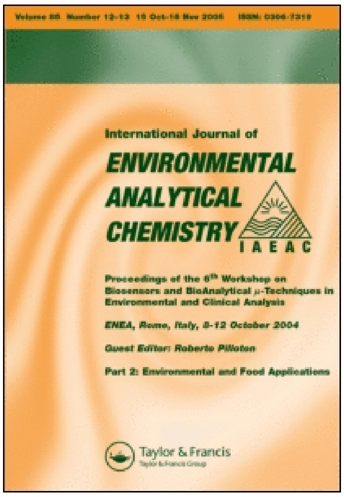

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Preparation, characterization and applications of polyurethane foam functionalized with resorcinol for quantitative separation and determination of silver(I) and mercury(II) from tap and wastewater

Elhossein Ali Moawed[a]; Mohamed Ahmed Zaid[b]; Mohamed Fathy El-Shahat[b]
[a] Department of Chemistry, Faculty of Science, Mansoura University, Damietta, Egypt [b] Department of Chemistry, Faculty of Science, Ain Shams University, Cairo, Egypt

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PREPARATION, CHARACTERIZATION AND APPLICATIONS OF POLYURETHANE FOAM FUNCTIONALIZED WITH RESORCINOL FOR QUANTITATIVE SEPARATION AND DETERMINATION OF SILVER(I) AND MERCURY(II) FROM TAP AND WASTEWATER

ELHOSSEIN ALI MOAWED[a,*], MOHAMED AHMED ZAID[b] and
MOHAMED FATHY EL-SHAHAT[b]

[a]*Department of Chemistry, Faculty of Science, Mansoura University, Damietta, Egypt;*
[b]*Department of Chemistry, Faculty of Science, Ain Shams University, Cairo, Egypt*

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A stable chelating resin matrix was prepared by covalently linking resorcinol with polyurethane foam matrix through a –N=N– group. Preconcentration and determination of trace Ag^+ and Hg^{2+} ions from samples of different origin, using Res-PUF, were studied. Various conditions influencing the sorption of these metal ions onto Res-PUF were optimized. The kinetics of sorption of the Ag^+ and Hg^{2+} by Res-PUF were found to be fast, reached equilibrium in few minutes (5–10 min) and followed a first-order rate equation with an overall rate constant k in 0.102 and 0.267/min, respectively. Study of the variation of the sorption of the tested metal ions with temperature yielded average values for ΔG, ΔH and ΔS of -3.94, -22.02 and -58.37, respectively. The mean free sorption energy (E) computed from the Dubinin–Radushkevich (D–R) isotherm was found to be equal to 8.91 kJ/mol, which reflects the chelation sorption process. The capacities of the foam material were 0.15 and 0.07 mmol/g for Ag^+ and Hg^{2+}, respectively. Preconcentration factors of >50 were achieved ($RSD \approx 5.99$). The proposed preconcentration procedure was applied successfully to the determination of trace metal ions in natural and wastewater samples.

Keywords: Silver (I); Mercury (II); Polyurethane foam; Resorcinol; Preconcentration

INTRODUCTION

During the last two decades, much attention has been paid to the development of chelating resins with high selectivity, high sorption capacity for metal ions, good stability and flexibility in working conditions. Two methodologies are frequently adopted for

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*Corresponding author. Fax: +966-3-826-9936. Email: eamoawed@yahoo.com

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such designing. The first method involves sorption of chelating ligands onto a matrix [1,2]. The second is based on covalent coupling of a ligand with the polymer backbone through a spacer arm, generally a –N=N– group. Several important and recently reported chelating matrices developed using such supports include Amberlite XAD-2 modified with pyrocatechol [3,4], chromotropic acid [4], thiosalicylic acid [4], quinalizarin [5], aminophenol [6], tiron [7], alizarin red s [8], salicylic acid [9], pyrocatechol violet [10] and *o*-vanillinthiosemicarbazone [11,12] through the –N=N– group.

The importance of polyurethane foam as collector material has increased due to its efficiency, low cost, easily handling and storage, which is convenient for field studies [13–21]. Recently, many reports have studied the chemical reaction of the functional group in the polyurethane foam by use of diffuse reflectance spectroscopy and infrared spectroscopy [22–26]. It was found that the functional groups are highly reactive towards oxidation by active chlorine [22], condensation with formaldehyde, diazotization by sodium nitrite, and azo coupling with 4-nitrophenyldiazonium tetrafluoroborate [23], 8-hydroxyquinoline [24], α-naphthol, β-naphthol [25] and alizarin red s [26]. Also, it has been found that the azo-derivatives formed were retained by the cation chelation mechanism. On this basis, an analytical methodology was developed in the present paper for the synthesis of a stable chelate-forming resin matrix by covalently linking resorcinol with polyurethane foam, which has been recycled many times without affecting its efficiency. This matrix has been studied for its use for the separation and preconcentration of silver(I) and mercury(II). The sorption mechanism of the metal ion complexes onto white polyurethane foam is also discussed.

EXPERIMENTAL

Reagents and Materials

All reagents were prepared from analytical reagent-grade chemicals and bidistilled water. A 1 mg/mL stock solution of Ag^+ was prepared by dissolving 0.1575 g of dried (110°C) $AgNO_3$ (Merck) in water containing 1 mL of conc. HNO_3 and dilution of the solution to 100 mL in a measuring flask. A stock solution containing 1 mg/mL of Hg^{2+} was prepared by dissolving 0.1713 of $Hg(NO_3)_2 \cdot H_2O$ (Merck) in water containing 1 mL of conc. HNO_3 and dilution of the solution to 100 mL in a measuring flask. A 1% resocinol solution was prepared by dissolving 0.1 g of reagent (Adwic) in 10 mL of 1 M NaOH. Commercial white sheets ($d \approx 20\,kg/m^3$) of open cell polyether type-based polyurethane foam was used.

Synthesis of the Res-PUF

Polyurethane foam functionalized with resorcinol was prepared. A portion of 5 g of small cubes ($\sim 0.125\,cm^3$) of white PUF cubes ($d = 20\,kg/cm^3$) was washed with distilled water, followed by acetone and allowed to dry at room temperature. The foam cubes were soaked in 50 mL of 3 M HCl stirred for 2 h, and cooled in an ice bath. To the foam cubes, 10 mL of 0.5 M $NaNO_2$ solution was added drop by drop with continuous stirring. The resorcinol solution was added to PUF, with stirring, and kept in the refrigerator for 2 h. Res-PUF material was filtered and then washed with 0.1 M HCl followed by distilled water, and finally with acetone before drying at room temperature.

Apparatus

All spectrophotometric measurements were performed using either Spectronic 501 (Milton Roy Company) or (UV-1601 (Shimadzu) spectrophotometers. Atomic absorption (Solaar 969 AA Spectrometer) was also used for determining Ag^+ and Hg^{2+} in real samples at 328.1 and 253.7 nm, respectively. The pH measurements were carried out using a pH meter from Microcomputer pH-vision. Glass columns about 15 cm long and 1.5 cm in diameter were employed in the chromatographic separation experiments.

GENERAL PROCEDURES

Sorption Investigations

Separation of Ag^+ and Hg^{2+} was carried out by a batch technique at 25°C except where otherwise specified. A 0.2 g portion of the Res-PUF was mixed with a 25 mL aliquot of a tested metal ion solution (1.6 μg/mL) in a shaker thermostatted to the desired temperature and adjusted to the desired shaking speed. After a certain time, the solution was separated, and the concentration of metal ion was determined spectrophotometrically by dithizone method in acidic medium [27].

Chromatographic Separations

In the dynamic experiments, 1 g of Res-PUF was packed into the column (15 cm × 1.5 cm) using the vacuum method of foam packing [13]. Test solutions were passed through the foam column at a flow rate of 3 mL/min. The stripping of the metal ions from the foam column was carried out with the eluting agent, and the amount of the metal ion was determined spectrophotometrically.

RESULTS AND DISCUSSION

Characterization of Res-PUF

The densities of the white PUF and Res-PUF were measured; the values obtained were 20 and 50 kg/m^3, respectively. From these results, it appears that the Res-PUF is denser than the white PUF. The higher density of the Res-PUF could be attributed to the inter-bonding between the reagents and the PUF groups.

The chemical stability of Res-PUF was studied, and the leaching of the reagent from Res-PUF with different solvents in batch mode was tested. The reagent was not detected in the tested solvents by using $FeCl_3$ or CH_2O/H_2SO_4. From previous results, Res-PUF shows a good chemical stability in the presence 1–6 M H_2SO_4, 1–6 M HCl, ethanol, isopropanol, butanol, chloroform, carbon tetrachloride and acetone. Also, the Res-PUF was stable for several months if stored in the polyethylene bag. These results indicate that the amount of resorcinol on the PUF matrix is 0.182 mmol/g. This was confirmed by the elemental analysis of the white and Res-PUF (Table I). The percentage of carbon, hydrogen and nitrogen of the Res-PUF was increased, and the percentage of oxygen was decreased due to the partial hydrolysis of some isocyanate and urethane groups (release CO_2) and coupling of the resorcinol (Fig. 1).

TABLE I Comparison between white PUF and Res-PUF

Property	*White PUF*	*Res-PUF*
Density, kg/m^3	20	50
UV/Vis. spectra	All band in UV specially at 280 nm	New band appear at 385 nm and shift to 400 nm after coupling
IR spectra		
–NH	Band at 3354.4/cm	Shift to 3381 and to 3458.6/cm
–NCO	Free isocyanate group at 2290/cm	Disappear
–N=N–	Nil	New bands at 1726.5 and 1549.4/cm
Elemental analysis		
C, H, N	62.84, 8.26 and 8.23%, respectively	64.91, 8.50 and 8.48%, respectively

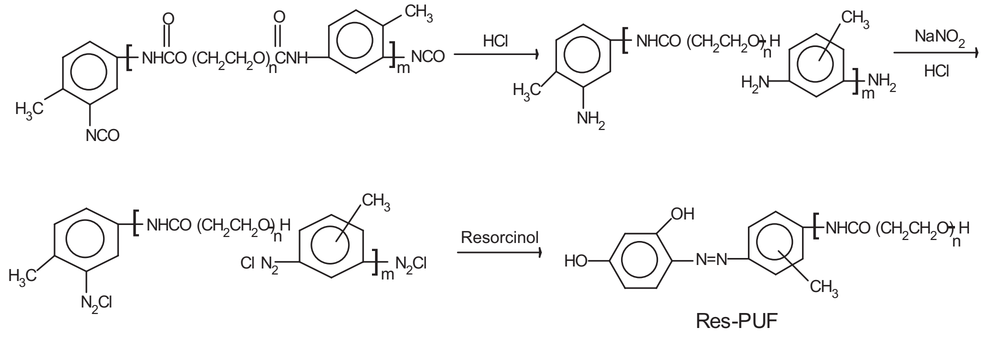

FIGURE 1 Scheme for the synthesis and the structure of resorcinol bonded polyurethane foam.

The results of elemental analysis suggest that an average of one resorcinol molecule is present in the Res-PUF per repeat unit of polymer.

For spectrophotometric measurements, the thin film of dry foam (2.5 × 0.8 × 0.2 cm) was placed in the path of light in the cell which was filled with ethanol. The UV-Vis spectrum of Res-PUF was compared with that of white PUF. The Res-PUF had a new absorption band at 385 nm after addition of $NaNO_2$ and shifted to 400 nm after coupling with resorcinol.

IR spectra of white and Res-PUF were studied using the potassium bromide technique. The results obtained show that in the Res-PUF spectrum, there was a shift in the absorption band of –NH of urethane groups (–NHCOO–) from 3354 to 3381/cm after washing with 3 M HCl and to 3458/cm after coupling with resorcinol, while the band at 2274.4/cm characteristic of isocyanate (–NCO) group of white foam disappeared. However, two additional bands at 1726.8 and 1549.4/cm characteristic of the Res-PUF appeared.

The capacity of the Res-PUF was calculated. The values obtained for Ag^+ and Hg^{2+} were 0.15 and 0.07 mmol/g Res-PUF, respectively, using the batch technique, and the corresponding molar ratios (M : Res-PUF) for these ions are 1 : 1.2 and 1 : 2.4, respectively. The capacity sequence was in the order $Ag^+ > Hg^{2+}$, and the difference in sorption capacities of these metal ions may be attributed to their different ionic sizes. The sorption capacity of Res-PUF is compared with Amberlite XAD-2 functionalized with different reagents [3–12], and the Res-PUF exhibits better or comparable capacity values in comparison with most of metal ions. The characterization of white PUF and Res-PUF is summarized in Table I. The expected scheme for the hydrolysis of some urethane and isocyanate groups of white polyurethane foam and interaction of foam group (–NH_2) with resorcinol for preparing Res-PUF is shown in Fig. 1.

Optimum Conditions for Preconcentration of Ag^+ and Hg^{2+} Using Res-PUF

Effect of pH on Extraction of Ag^+ and Hg^{2+}

In batch experiments, an aliquot of the aqueous working solution (4 µg/mL) of Ag^+ and Hg^{2+} each alone was shaken with 0.2 g of the Res-PUF at various pHs. Their pH values were adjusted in the range 1–12 with 0.1–1 M HNO_3/NaOH. The pH values were plotted against $\log K_d$, as shown in Fig. 2. The optimum pH ranges to extract Ag^+ and Hg^{2+} from aqueous solution were 1–5 and 1–4, respectively. The selectivity sequence was $Ag^+ > Hg^{2+}$ in the pH range 1–12. These results show that controlling the pH can play a role in enhancing the selectivity of the tested metal ion separation with Res-PUF. Also, the values of the distribution coefficient decrease with increasing pH values ($\log K_d = \text{constant} - n\text{pH}$).

Kinetic Study

Kinetic measurements were carried out using Res-PUF with a diameter of 0.5 cm and V/M of 125 mL/g. The amount of Ag^+ and Hg^{2+} extracted on Res-PUF was measured by batch extraction at different time intervals, i.e. 1–60 min. From the results obtained, the time required for sorption equilibrium was found to be 5–10 min (Fig. 3).

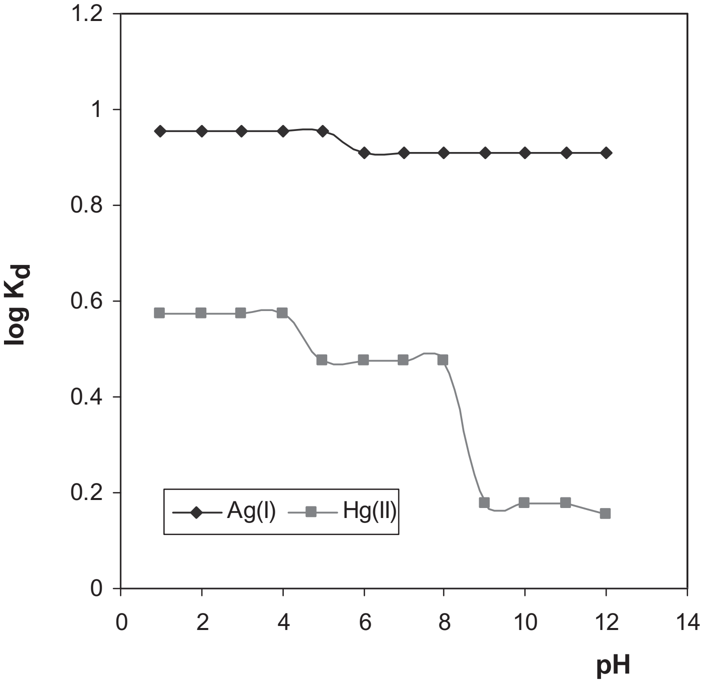

FIGURE 2 Effect of pH on the distribution coefficient for the sorption of Ag^+ and Hg^{+2} onto Res-PUF.

TABLE II Kinetic parameters for the sorption and desorption of metal ions onto Res-PUF

Metal ions	*Rate constant of sorption* k_1/min	*Rate constant of desorption* k_{-1}/min	*Overall rate constant* k'/min	*Half-life of sorption* $t_{1/2}$/min	*Diffusion rate of sorption* k_M μmol/g min
Ag^+	0.102	0.011	0.113	6.26	1.44
Hg^{2+}	0.267	0.071	0.338	2.39	1.14

TABLE III Average thermodynamic parameters of the sorption of Ag^+ and Hg^{+2} onto Res-PUF at different temperature

Metal ions	*Gibbs free energy* ΔG (kJ/mol)	*Enthalpy* ΔH (kJ/mol)	*Entropy* ΔS (J/K mol)	*Activation energy* ΔE (kJ/mol)
Ag^+	−5.07	−24.03	−61.93	8.81
Hg^{2+}	−2.82	−20.00	−54.67	9.01

In order to study the particle diffusion mechanism, the Morris–Weber equation was applied. The data obtained show that the diffusion rate is rapid, and the values of k_M are 1.44 and 1.14 μmol/g min$^{1/2}$ for sorption of Ag^+ and Hg^{2+}, respectively (Table II).

The kinetic parameters for sorption of the tested metal ions onto the Res-PUF according to a first-order reaction and a time-dependent process are given by the Lagergren equation. A straight-line plot of $\log(q_{e-}q_t)$ vs. t indicates that the process is a first-order reaction. The rate constants for the sorption, calculated from the slope, are 0.102 and 0.267/min for Ag^+ and Hg^{2+}, respectively. The half-life of sorption, rate constant of desorption, and the overall rate constant at room temperatures were then calculated. The values of $t_{1/2}$, k_1, k_{-1}, and k' are summarized in Table II; the short loading time ($t_{1/2}$) of the Res-PUF shows that the rapid extraction is due to the application of the batch technique, which is relatively fast and efficient for the Res-PUF sorbent as compared with the other resins [28–30].

Thermodynamic Investigations

The dependence of sorption of Ag^+ and Hg^{2+} ions on Res-PUF with temperature has been evaluated using the equations $\Delta G = -RT \ln K_c = \Delta H - T\Delta S$, where ΔG, ΔH, ΔS, and T are the Gibbs free energy, enthalpy, entropy, and absolute temperature, respectively, R is the gas constant (8.314 J/mol), and K_c is the equilibrium constant. The values of ΔG, ΔH and ΔS at different temperatures for the sorption of Ag^+ and Hg^{2+} are given in Table III. The negative values of ΔG are attributed to the spontaneous nature of the sorption process. Similarly, the negative values of ΔH may be interpreted as indicative of exothermic chemosorption processes, while a negative change in the entropy may be indicative of the faster sorption of Ag^+ and Hg^{2+} onto Res-PUF.

Effect of Metal Ion Concentration

Uptake of the metal ions on the Res-PUF was determined as a function of metal ion concentration in the aqueous solution (Fig. 4). The resultant isotherms show a good linear relationship over a relatively wide range of tested ion concentrations. The effects of tested metal ion concentrations on Res-PUF were analyzed in terms of the

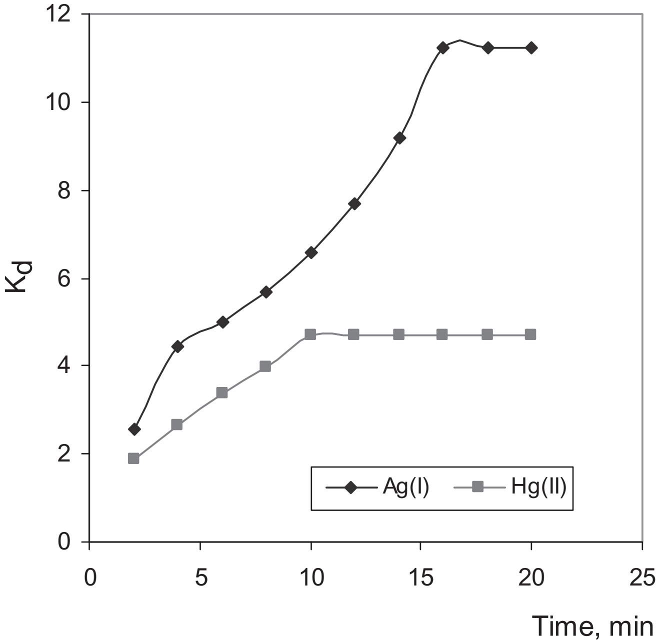

FIGURE 3 Effect of shaking time on the extraction of Ag^+ and Hg^{+2} onto Res-PUF.

Freundlich, Langmuir, Misak, and Dubinin–Radushkevich equations. The plot of q_e/q_c vs. q_e for the experimental data according to Langmuir and Misak models does not give a linear relationship. These models suggested that sorption of metal ion from solution by the solid is a monolayer coverage. The plot of $\log q_c$ vs. $\log q_e$ shows a straight line, and the values of Freundlich constants $(1/n)$ from the slope were 0.96 and 0.94, which may be attributed to the heterogeneous surface structure of the Res-PUF. The Dubinin–Radushkevich isotherm was tested. The values of β for sorption of Ag^+ and Hg^{2+} computed from the slope of the linear plot of $\ln q_c$ vs. ε^2 are -6.44×10^{-3} and $-6.19 \times 10^{-3}\,kJ^2/mol^2$, respectively. The value of the sorption energy (activation energy, E) can be correlated with β ($E = 1/\sqrt{-2\beta}$). The values of E evaluated are 8.81 and 9.01 kJ/mol for Ag^+ and Hg^{2+}, respectively, indicating that the rate of sorption is relatively fast.

Breakthrough Capacity

From the breakthrough curves presented in Fig. 5, the saturation of each column was reached after passage of 45–50 mL. The height equivalent to a theoretical plate (HETP) was calculated from the breakthrough curves by using the equation:

$$\mathrm{HETP} = \frac{L(V - \overline{V})^2}{V\overline{V}}$$

where V is the volume of effluent at the center of the S-shaped breakthrough where the concentration is one-half the initial concentration, and $\overline{V}$ is the volume at

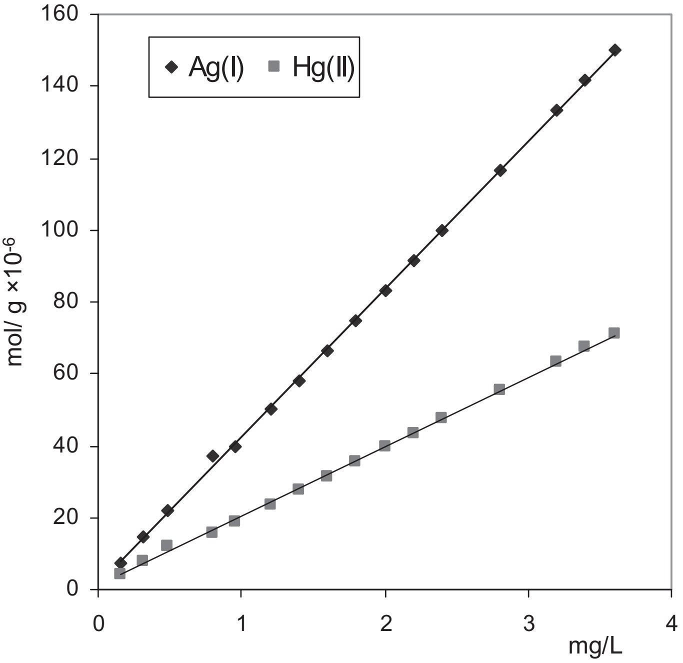

FIGURE 4 Isotherm curve of the sorption of Ag^+ and Hg^{+2}.

which the effluent has a concentration of 0.1587 of the initial concentration. The values of HETP obtained using this equation were 0.25, and 0.67 mm for Ag^+ and Hg^{2+}, respectively.

Effect of the Sample Flow Rate

The sorption of Ag^+ and Hg^{2+} on the Res-PUF column was studied at different solution flow rates. For each metal ion, a set of solutions (100 mL) containing 40 μg were passed through the Res-PUF column at a flow rate varying from 1 to 7 mL/min. The metal ions could be eluted by 0.1 M HNO_3 and determined spectrophotometrically by dithizone method in acidic medium. It was found that the optimum flow rate was 2 mL/min. The percentage uptake decreased from 95 to 90 by increasing the flow rate from 2 to 7 mL/min.

Preconcentration of Metal Ions from Different Solution Volumes

The effect of the ratio of sample volume to the elution volume (preconcentration factor) on the percentage uptake was studied; the results obtained are shown in Fig. 6. Elution of Ag^+ and Hg^{2+} from Res-PUF columns was achieved with recoveries of 80–98% (average RSD $\approx$ 5.99). The percentage uptake of the metal ions decreased (from 98% to 80%) with increasing sample volume (0.05 $\rightarrow$ 1 L). These results show that the tested metal ions can be concentrated effectively from large volumes of dilute aqueous solutions using Res-PUF columns. Preconcentration factors (P) above 50 were achieved, and the detection limit (LDL) was found to be lower than 0.4 μg/L for

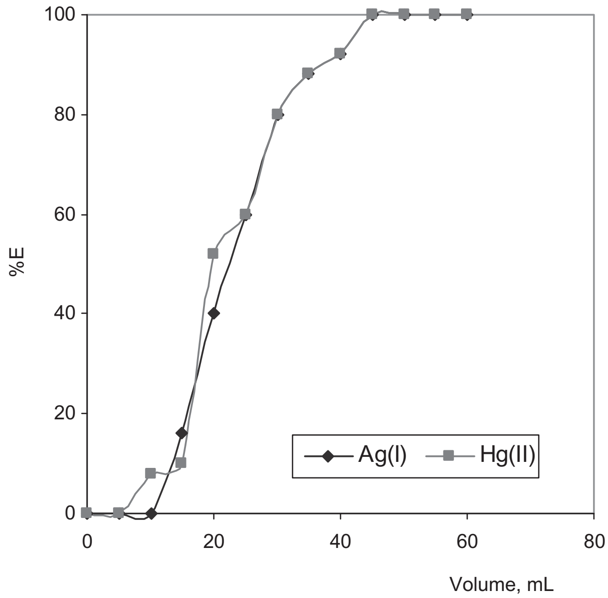

FIGURE 5 Breakthrough curve for extraction of Ag^+ and Hg^{+2} from aqueous solution.

each element. These values of P and LDL are in good agreement with those obtained using Amberlite XAD-2 [3–12].

Effect of Foreign Ions

The effect of different Na^+, K^+, Mg^{2+}, Ca^{2+}, Ba^{2+}, Zn^{2+}, Cd^{2+}, Al^{3+}, Fe^{2+}, Fe^{3+}, Co^{2+}, Cu^{2+} and Ni^{2+} (40 μg/mL) on the sorption of Ag^+ and Hg^{2+} (1.6 μg/mL) onto Res-PUF was studied using a batch technique. The obtained data reveal that Na^+, K^+, Mg^{2+}, Ca^{2+} and Ba^{2+} do not have any adverse effects on the sorption of Ag^+ and Hg^{2+}, while Zn^{2+}, Cd^{2+} and Al^{+3} interfere slightly (reduce the sorption 1–3%). Also, Fe^{2+}, Fe^{+3}, Co^{2+}, Cu^{2+} and Ni^{2+} reduce the sorption of tested metal ions 10–20%. Besides controlling the pH of the sorption medium, a masking agent such as tartaric acid, citrate or phosphate ions masks several common ions and minimizes their interfering effect.

Metal Ion Separation

A mixture of Ag^+ and Hg^{2+} ($pH \approx 3$) was passed through the Res-PUF column at a flow rate of 2 mL/min. Ag^+ was eluted with 0.1 M NH_3, and finally Hg^{2+} was eluted with 4 M NH_3 (Fig. 7). This technique enabled selective determination of each metal ion from the other ion in both mixture.

Collection and Determination of Ag^+ and Hg^{2+} from Tap and Wastewater Using Res-PUF

The analytical applicability of the proposed Res-PUF was tested for collection of Ag^+ and Hg^{2+}. Ag^+ and Hg^{2+} in a certified reference tap water sample (Central

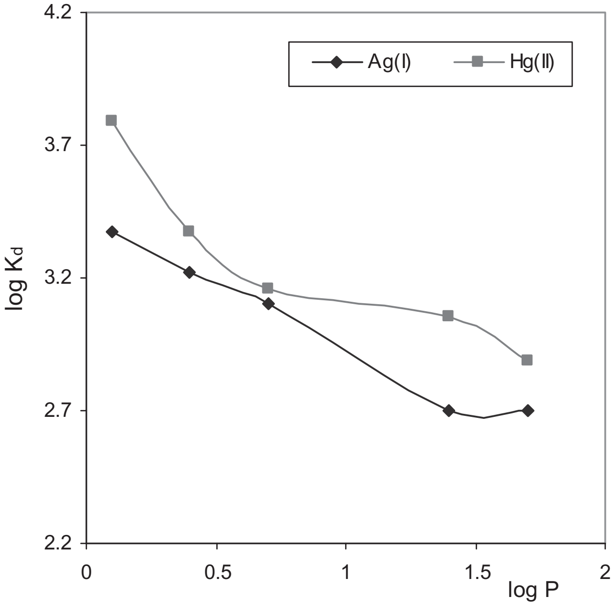

FIGURE 6 Effect of preconcentration factor (P) on distribution ratio for sorption of Ag^+ and Hg^{+2} from aqueous solution on to Res-PUF.

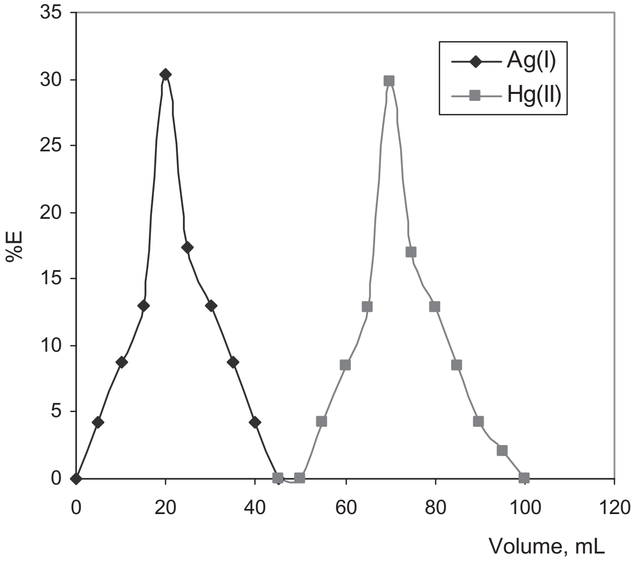

FIGURE 7 Separation of Ag^+ and Hg^{+2} from aqueous solution using a Res-PUF column and 0.1 M NH_3 and 4 M NH_3 as eluting agents.

Organization Greater Cairo of Water Supply 'GOGCWR' has been certified ISO 9001/2000) were determined using a dynamic method. The results obtained (Ag^+ and Hg^{2+} were nil) are in agreement with the values obtained using AAS measurements in the central laboratory of GOGCWR. Also, industrial wastewater samples from different

TABLE IV Collection of Ag^+ and Hg^{+2} from tap[a] and wastewater[b] using Res-PUF

Sample	*Ag^+*			*Hg^{+2}*		
	Add (μg)	*Recovery* (%)	*RSD*	*Add* (μg)	*Recovery* (%)	*RSD*
Central organization of Greater Cairo water supply	10	90		10	100	
Wastewater of superphosphate units	15	90	3.38	10	100	5.00
Wastewater of sulphuric acid unit	5	90		20	100	
Cooling pond of phosphoric acid unit	5–12.5	≈83.3		5–12.5	≈90	

[a] Tap water composition: Na 29.24 mg/L; K 5.1 mg/L; Cr 0.17 μg/L; Ni 1.65 μg/L; Fe 0.088 mg/L; Mn 0.008 mg/L, pH 7.2.
[b] Wastewater composition: P_2O_5 4 g/L; SO_4^{2-} 0.3 g/L; H_2SiF_6 2.5 g/L; Fe^{3+} 25 mg/L; Zn 8 μg/L; Cd 2 μg/L, pH 2–6.8.

positions (cooling pond of the phosphoric acid production, wastewater of superphosphate and wastewater of sulphuric acid units) of Abu-Zaable Fertilizers and Chemicals Company (ABZFC) and the tap water sample of GOGCWR were used to study the adsorption efficiency and the yield recovery of Ag^+ and Hg^{2+} from the resin. A 100 mL aliquot of wastewater was spiked by standard addition (5–25 μg) of Ag^+ and Hg^{2+} at the adjusted pH. The solutions were then passed through the columns at a flow rate of 2 mL/min. The metals were eluted with 5 mL of 1 M HNO_3 and determined using AAS. The results are given in Table IV, which shows the suitability of the Res-PUF for water analysis and the removal of the metal ions from industrial wastewater.

CONCLUSION

This work deals with the preparation of a new polymeric extractor based on covalent coupling of resorcinol with polyurethane foam through an azo group. This new extractor (Res-PUF) was used to preconcentrate and separate Ag^+ and Hg^{2+} from aqueous solutions. Characterization of the Res-PUF indicates that it is denser than the white foam and has more cross-linkage due to the additional bonds between the reagent and the other groups in the polyurethane foam matrix. It has been recycled many times after regenerated using 1 M HNO_3 and NaOH without affecting its efficiency. The kinetics of sorption of the tested metal ions on to Res-PUF follow a first-order rate equation. The negative values of ΔG indicate the spontaneous nature of the sorption of the tested metal ions. An exothermic reaction may be assumed due to the negative values of ΔH for the sorption of tested metal ions. The method was successfully applied for the retention and recovery of the Ag^+ and Hg^{2+} spiked to tap and industrial wastewater. In comparison of Res-PUF with white-PUF (unloaded PUF) and PUF impregnating (physical loading) with ligands (loaded PUF) via crystal violet [17,19,21] for sorption of Ag^+ and Hg^{2+}, the Res-PUF has a better sorption capacity and lower detection limit than loaded and unloaded PUF due to the leaching of ligands from the loaded PUF and the lack of selectivity of unloaded PUF. Along with its stability, the Res-PUF has the additional advantage in that there is virtually no change in the sorption capacity of the resin, even after it has been used many times.

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